High-Cycle Fatigue of Single-Crystal Silicon Thin Films

Christopher L. Muhlstein, Stuart B. Brown, and Robert O. Ritchie

***Abstract*—When subjected to alternating stresses, most materials degrade, e.g., suffer premature failure, due to a phenomenon known as *fatigue*. It is generally accepted that in brittle materials, such as ceramics, fatigue can only take place in toughened solids, i.e., premature fatigue failure would not be expected in materials such as single crystal silicon. The results of this study, however, appear to be at odds with the current understanding of brittle material fatigue. Twelve thin-film ($\sim$ 20 μm thick) single crystal silicon specimens were tested to failure in a controlled air environment (30 $\pm$ 0.1°C, 50 $\pm$ 2% relative humidity). Damage accumulation and failure of the notched cantilever beams were monitored electrically during the "fatigue life" test. Specimen lives ranged from about 10 s to 48 days, or 1×10^6 to 1×10^{11} cycles before failure over stress amplitudes ranging from approximately 4 to 10 GPa. A variety of mechanisms are discussed in light of the fatigue life data and fracture surface evaluation. [642]**

***Index Terms*—Fatigue failure, MEMS devices, single-crystal silicon, thin films.**

I. Introduction

THE application of common MEMS, such as pressure transducers, inertial sensors, and ink jet cartridge nozzles, has already resulted in significant improvements in performance, and reductions in cost, for many industries ranging from medical device manufacturing to computing. Bolstered by this success, manufacturers are developing micromechanical components made of silicon-based structural films in actuator, power generation, and other "safety-critical"[1] and "high-performance" applications. However, these safety-critical structures are often subjected to aggressive mechanical and chemical environments without sufficient understanding of the behavior of the material under such conditions; this is especially pertinent as the dimensions of the material components may be far smaller, i.e., micrometer-scale and below, than has been conventionally tested in mechanical property evaluations. Consequently, in order to ensure performance and reliability, design approaches must be employed that account for the time- and cycle-dependent degradation of the material at the size-scales of interest.

Micromechanical components are routinely subjected to cyclic stresses at kilo- and megahertz frequencies, accumulating large numbers of stress cycles in relatively short periods of time. It is important to note that when cyclic stresses are applied, most materials degrade, e.g., suffer premature failure, due to a phenomenon known as *fatigue.* Fatigue is the most commonly experienced form of structural failure, yet surprisingly it is one of the least understood. The most well known form of fatigue, the cyclic fatigue of metals, is generally associated with the generation and motion of dislocations and the accumulation of plastic deformation; these processes can lead to the creation and advancement of a nucleated or preexisting crack by alternately blunting and sharpening the crack tip (striation formation). However, the corresponding mechanisms of fatigue in brittle materials, such as the structural films commonly used in MEMS, are quite different. Due to their high Peierls forces, brittle materials such as ceramics and single crystal silicon have very limited dislocation mobility at low homologous temperature, making the possibility of cyclic fatigue failure far less obvious. However, premature cyclic fatigue can occur in brittle materials, e.g., polycrystalline ceramics and ordered intermetallics, but by a conceptually different mechanism [1], [2]. Such failures are generally associated with kinematically irreversible deformations; specifically, crack-tip shielding mechanisms that operated primarily in the crack wake and are the basis of their fracture toughness tend to degrade under cyclic loading conditions.[2] However, this mechanism can only take place in toughened solids where there is some degree of shielding to degrade; it would not be expected in materials such as single crystal silicon.

Despite this, studies of both mono- and polycrystalline silicon thin films have established that crack growth can occur in room-temperature air environments under cyclic loading conditions. [3], [4] In both cases, water vapor was demonstrated to play an important role in the growth of intentionally introduced cracks in cantilever beam resonators. Although water vapor is often associated with enhanced crack growth under static and cyclic stresses in both ductile and brittle materials, most studies have indicated that bulk, single crystal silicon is not susceptible to either cyclic fatigue or environmentally assisted (e.g., stress–corrosion) cracking [5]–[10]. Two possibilities may account for the discrepancy between the bulk and thin-film studies. First, the bulk tests cannot resolve the low crack-growth rates

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[1]The term "safety-critical" application is used here to imply applications where mechanical failure of the device or structure could result in loss of human life.

[2]An example of this is the cyclic fatigue of polycrystalline ceramics, such as Al_2O_3, Si_3N_4, and SiC, which fail intergranularly and are thus toughened by the bridging of interlocking grains in the crack wake. Under cyclic loading, frictional wear in the sliding interfaces can lead to a progressive degradation in the potency of these grain bridges, thereby effectively "detoughening" the material (e.g., [1]).

relevant to micromechanical systems, where incipient cracks may be propagating due to a stress–corrosion mechanism but at extremely low velocities. Second, a true cyclic fatigue effect (via an as yet undefined mechanism) may be responsible. While investigations of the mechanism(s) of crack advance in silicon thin films are currently in progress, we can gain important insights into the mechanisms of crack initiation in these materials fatigued in room-temperature air environments.

Considerations of fatigue-crack initiation and growth must be a crucial part of critical component design, since the majority of the lifetime of a structural component will invariably be spent in the initiation and early growth of small flaws. As most mechanical components contain notches, the process of fatigue-crack initiation and small crack behavior in the presence of notches is a critical feature to be understood. Previous studies of polycrystalline silicon by two of the authors have shown that delayed failure can occur under cyclic stresses [3], [4], [11], [12]; these observations have been recently confirmed [13]. The objective of this study is to investigate the susceptibility of single crystal silicon to the growth of flaws under cyclic loading in the vicinity of a lithographically patterned, micromachined notch.

II. Experimental Methods

A. Test Techniques

Fatigue-crack initiation studies are typically conducted per the recommendations of ASTM Standard E 466. This standard addresses appropriate specimen design and techniques for the stress/strain-life (*S*/*N*) testing of bulk metallic materials. In fatigue-crack initiation testing, specimens are usually subjected to uniaxial, cyclic forces until a specified number of cycles elapse, a preselected plastic strain (i.e., 1%) accumulates, or a complete separation of the specimen occurs. The stress or strain amplitude may be the selected controlling parameter for the test depending on the behavior of the material, specifically the extent of plastic deformation over the load range of interest. The mean and amplitude of the stress or strain range may be varied to probe various aspects of the material's behavior or to reflect actual service conditions. The time to failure, or "life" of the specimen, is then represented as a function of the range of applied cyclic stress or strain (the *S*/*N* curve). Provided that the specimen has been properly designed and that allowance is made for such aspects as safety factors, the presence of notches, environmental effects, and so forth, these data may be used to estimate component structural lifetimes and to minimize the risk of fatigue failures in service.

Although ASTM E 466 and other accepted fatigue life characterization standards are not directly applicable to micrometer-scale testing, the philosophy of specimen design and testing methodology established in these standards provides useful guidance when developing new characterization techniques. Fatigue specimens must be designed to fail in the gauge section under well-defined loading conditions. Furthermore, material anisotropy, residual stresses, and processing limitations must also be considered. Finally, in addition to the size effects, the actual service conditions of the engineering components must be considered to ensure that the fatigue life

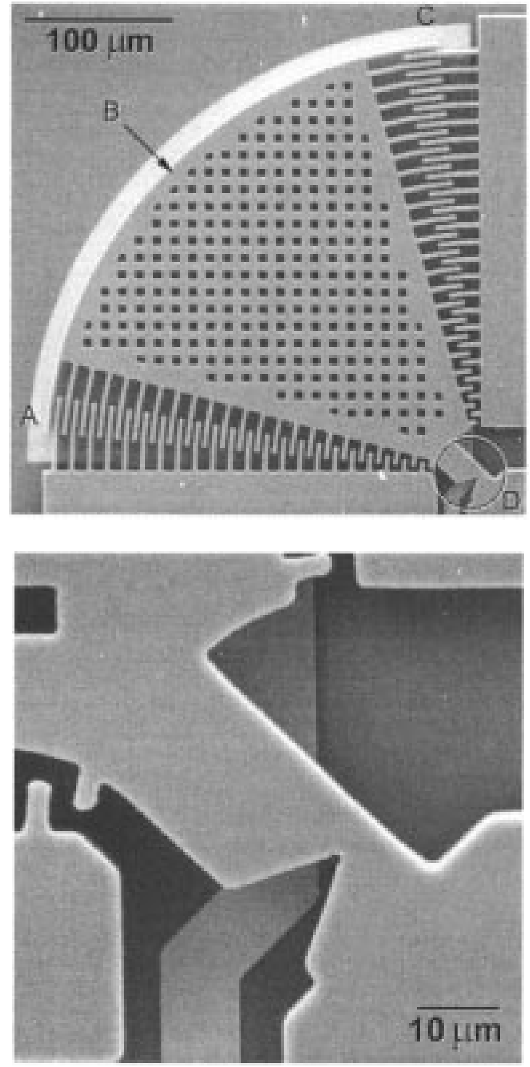

Fig. 1. Scanning electron micrographs of the stress-life fatigue characterization structure. The electrostatic comb drive actuator (A), resonant mass (B), capacitive displacement transducer comb (C), and notched cantilever beam specimen (D) are shown in an overview on the top. A detail of the notched beam is shown on the bottom.

data may be used for remaining life predictions and engineering design. The extreme difference in length scale between MEMS and conventional structural components is the impetus for testing thin film rather than bulk samples and the development of this test technique.

Over the past decade, starting at the Massachusetts Institute of Technology and continuing at Exponent Inc. and the University of California at Berkeley, we have developed a specimen geometry and "test structure" for characterzation of fatigue-crack initiation in thin films and have explored fatigue-crack initiation in a variety of materials systems. The "structure" design is based on the philosophy that underlies standards such as E 466. The micrometer-scale fatigue characterization structure shown in Fig. 1 is approximately 300 μm^2. This structure is analogous to a specimen, electromechanical load frame, and capacitive displacement transducer found in a conventional mechanical testing system. The specimen is a notched cantilever beam that is in turn attached to a large, perforated plate that serves as a resonant mass. The mass and beam are electrostatically forced to resonate and the resulting motion is measured capacitively. On opposite sides of the resonant mass are interdigitated "fingers" commonly referred to as "comb drives." One side of these drives is for electrostatic actuation; the other side provides capacitive sensing of motion. The specimen is attached to an electrical ground, and a sinusoidal voltage at the appropriate frequency is applied to one comb drive, thereby inducing a resonant response in the plane of the figure. The opposing comb drive is attached to a constant potential difference, and the relative motion of the grounded and biased fingers induces a current proportional to the amplitude of motion. The small induced current is converted to a direct current (dc) voltage using a circuit containing transimpedance amplifiers. A wide variety of capacitive sensing strategies can be found in the literature [14]. The output of the circuit used in this study was calibrated using the computer microvision system developed by Freeman. [15] Photolithography is used to introduce a stress concentration, as shown in Fig. 1. The radius of the stress concentration and the remaining beam ligament were selected to ensure that the specimen could be broken immediately at resonance. The longer term fatigue response can then be measured by exciting the specimen at some fraction of the short time breaking amplitude. All samples were tested until failure occurred by fracture of the beam at the notch, as shown in Fig. 2.

Given the well-established properties of single crystal silicon and the known displacement amplitude, the stress amplitude at the notch could be determined. An analytical cantilever beam model combined with stress concentration factors derived from finite-element methods [16] were used to calculate the applied stress amplitude. Although this approach neglects the effect of the elastically compliant anchor, it does provide a conservative estimate of the stress amplitude at the notch. Finite-element models of a cantilever without a notch indicate an upper bound of 5% error due to the compliance of the anchor.

The resonant frequency is used to monitor the accumulation of fatigue damage in the specimen until failure occurs at the notch. The device is driven at resonance using the following control scheme: The first mode resonant response of the specimen is determined by sweeping a range of frequencies around the expected response and monitoring the output of the sense circuit. The peak output is selected by fitting a second-order polynomial to the peak and extracting the maximum. The specimen is then excited at the peak frequency at a defined excitation voltage for a period of time. The frequency response is then again evaluated by sweeping around the excitation frequency. Over time, this permits measuring any change in resonant frequency and consequently any change in the mechanical response of the specimen. This scheme is simple yet effective in detecting the compliance changes associated with crack growth and damage accumulation.

B. Materials and Test Samples

In this study, p-type (110) single crystal silicon samples were photolithographically patterned and bulk micromachined using deep reactive ion etching. The silicon had the purity typical of electronic grade, single crystal silicon. Typical mechanical properties of single crystal silicon are readily available in the literature. The elastic modulus of 168.9 GPa

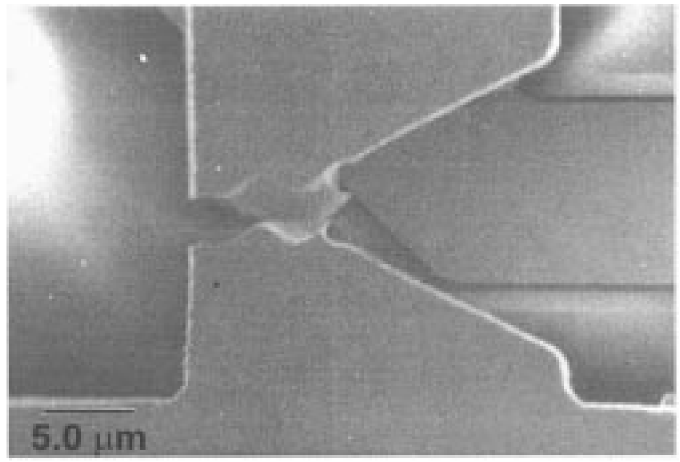

Fig. 2. Notch region of a failed thin-film single crystal silicon fatigue specimen.

in the $\langle 110 \rangle$ direction was calculated based on the compliance matrix of single crystal silicon [17], [18]. The strength of the bulk and thin-film single crystal silicon has been reported to vary from 1 to 20 GPa depending on the specimen type, size, preparation, and test technique [19]–[23]. Measured values of the fracture toughness of single crystal silicon range from 0.7 to 1.3 MPa$\sqrt{\text{m}}$ depending on specimen type, orientation, and investigator [5], [9], [19], [24]–[27],. The two groups of notched beams in this study have been designed for testing at approximately 40 and 50 kHz. The resulting notched beams were approximately 40 μm long, 21.5 μm wide, and 20 μm thick. The notch was located 8.1 μm from the base and was 14.3 and 13.3 μm deep for the 40- and 50-kHz resonant frequency specimens, respectively. The notch root radius was approximately 1 μm and represents the smallest root radius that could be created given the processing conditions. The beams were oriented along the $\langle 110 \rangle$ direction, and thus the maximum principal stress acted on the (110) plane.

III. Results and Discussion

Twelve thin-film (20 μm thick) single crystal silicon specimens were tested to failure in a controlled air environment [$30 \pm 0.1\,^\circ$C, $50 \pm 2\%$ relative humidity (RH)] using the sample geometry and testing procedure described above. Specimens were allowed to resonate for 1 to 5 min followed by recharacterization of the resonant frequency. The notched beams were driven in the in-plane bending resonant mode with a sinusoidal waveform from up to 95 V_{rms} with no DC offset. These conditions generated fully reversed stresses at the notch, i.e., a load ratio (minimum load by maximum load) of $R = -1$. Changes in resonant frequency were monitored by sweeping the drive frequency and recording the amplitude response using the previously described procedure. The "fatigue life" test duration ranged from about 10 s to 48 days, or 1×10^6 to 1×10^{11} cycles before failure over stress amplitudes ranging from approximately 4 to 10 GPa. This stress amplitude was controlled to better than 1% accuracy. Upon completion of the test, the measured lifetimes, the compliance change, and the resulting fracture surfaces were evaluated.

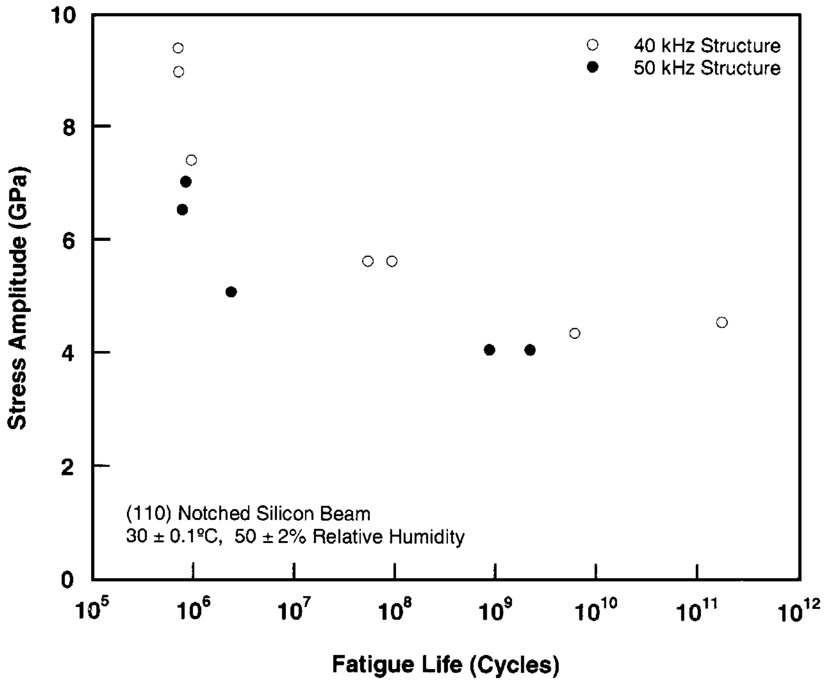

Fig. 3. Stress-life (*S*/*N*) fatigue curve of thin-film single crystal silicon in air (30 ± 0.1 °C, 50 ± 2% relative humidity). Tests conducted at nominal resonant frequencies of 40 and 50 kHz are shown.

A. Specimen Life and Damage Accumulation

The thin-film silicon cantilever beams exhibited a time-delayed failure under fully reversed, cyclic stresses in room-temperature air with 50% relative humidity. The life of the two notched beam configurations as a function of fully reversed stress amplitude is shown in Fig. 3. One would expect the peak tensile stress in the short life tests to approach the single cycle, or ultimate, strength of the material. Assuming this is the case, the peak stresses measured during the short life tests in the single crystal silicon used in this study fell within the, albeit wide, range of ultimate strengths measured for silicon. Typically, delayed failures in brittle materials in stress-life tests will be clustered around this stress level. The life of the thin-film silicon, however, increased monotonically with decreasing stress amplitude with a 50% reduction in stress amplitude, resulting in an increase in life of approximately five orders of magnitude; no significant effect of frequency between 40 and 50 kHz was observed. Of importance here is that unlike bulk silicon, these data imply that thin films of single crystal silicon can fail at stresses as low as one-half of their fracture strength when such stresses are applied cyclically in a moist environment for in excess of 10^{10} cycles. This trend has not been observed in the fatigue stress-life testing of any bulk brittle materials (e.g., [2]). Instead, a range of lives close to the strength of the material is observed, with few delayed failures at lower stress amplitudes.

The resonant frequency of the cantilever beam was used to monitor the specimen during cyclic loading in the controlled environment and was observed to decrease monotonically before the specimen finally failed at the notch. This strongly suggests that the failure of the thin-film silicon occurs after progressive accumulation of damage, e.g., by the stable propagation of a crack. This manifests itself as a progressive decay in the stiffness (resonant frequency) of the beam, as shown in Fig. 4. The longer the life of the specimen, the larger the decrease in beam stiffness (Fig. 5), which is again consistent with the notion of the accumulation of damage. However, it has proved to be difficult to correlate the growth of flaws observed on the fracture surfaces with such compliance changes.

B. Fractography

The surface morphology of a failed specimen can provide useful information about the initiation site and the failure mechanisms. The fracture surfaces of the single crystal silicon specimens were viewed using the scanning electron microscope (SEM) to provide this information as well as insight to the various damage processes. After an initial evaluation in the microscope, a thin layer of gold was sputtered onto the surface to improve image contrast. Typical fracture surfaces of specimens with short lives ($< 10^6$ cycles) and longer lives ($> 10^6$ cycles) are shown in Fig. 6. The area to the left of the fracture surface in the micrographs is the sidewall of the notch in the specimen. The horizontal banding and the vertical streaks in this region are a direct result of the deep reactive ion etch used to micromachine the component. The area to the right of the notch is the fracture surface of the notched beam. The difference in fracture surface morphology is quite striking and provides some important insight into the delayed failure of silicon.

The fracture surface of specimens with short lives revealed no clear initiation region and contained ridges, ledges, steps, and other features commonly observed on brittle material fracture surfaces, including silicon. Where possible, the orientation of the fracture surface was determined by trace analysis with respect to the $\langle 110 \rangle$ axis of the beam. In those cases where the orientation could not be measured, the general surface morphology was compared to fractography of bulk samples of the same sample orientation available in the literature [19], [24].

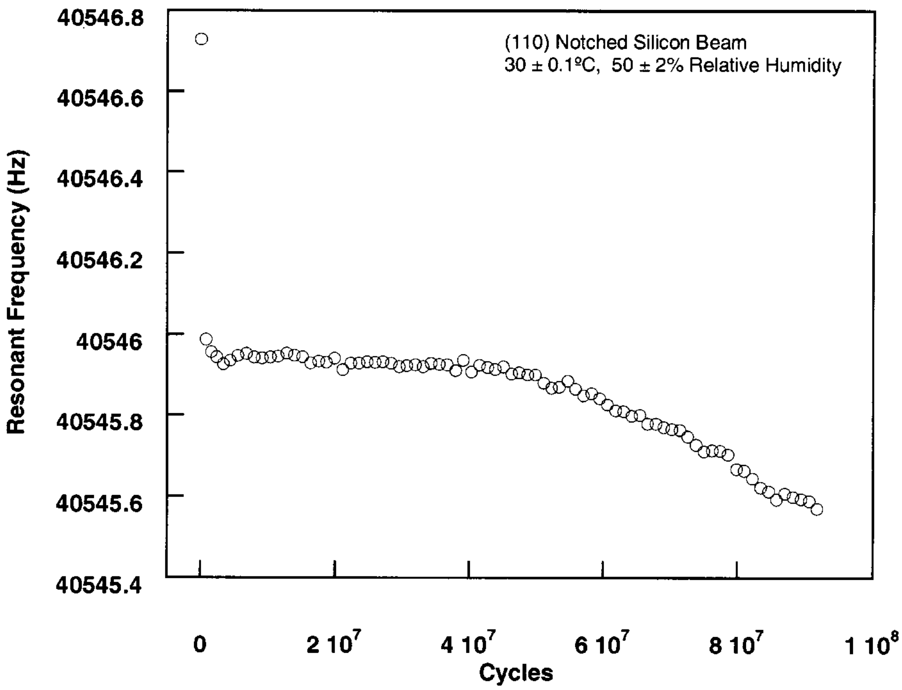

Fig. 4. Typical resonant frequency decrease during fatigue testing at 30 ± 0.1 °C, 50 ± 2% relative humidity.

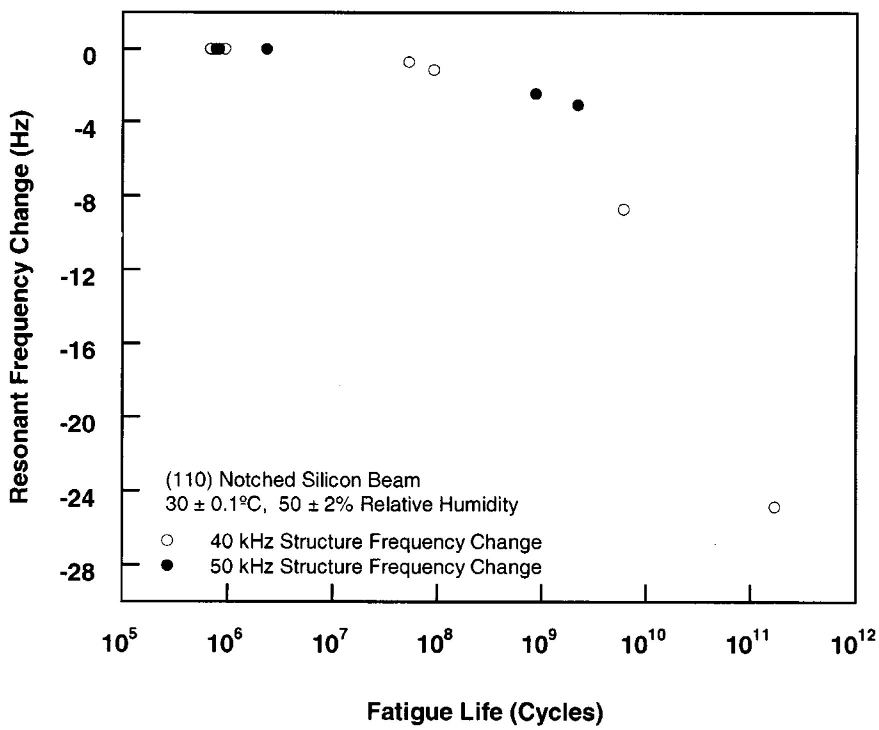

Fig. 5. Resonant frequency change as a function of specimen life in air (30 ± 0.1 °C, 50 ± 2% relative humidity).

The failure progressed by cracking on multiple {111} planes with final failure taking place by cleavage on a (111) plane after coalescence of multiple crack fronts. This transition in crack path manifests itself as an abrupt change in fracture surface morphology in the final third of the sample shown in Fig. 6(a). Because of the lack of both room-temperature plasticity and large volume defects in single crystal silicon, the dominant mode of failure is cleavage initiated at, or near, the surface of the notch. The morphology of the silicon fracture surfaces is a direct result of the crystallography of this cleavage. Members of the {111} planes are the cleavage planes for diamond cubic materials such as single crystal silicon. Cleavage steps form on {111} fracture surfaces as a means of dissipating energy during failure. These steps may form due to perturbations in the applied loads, obstacles in the crack path, bifurcation of fast running cracks, or the coalescence of microcracks [28]. Flaws that initiate on higher

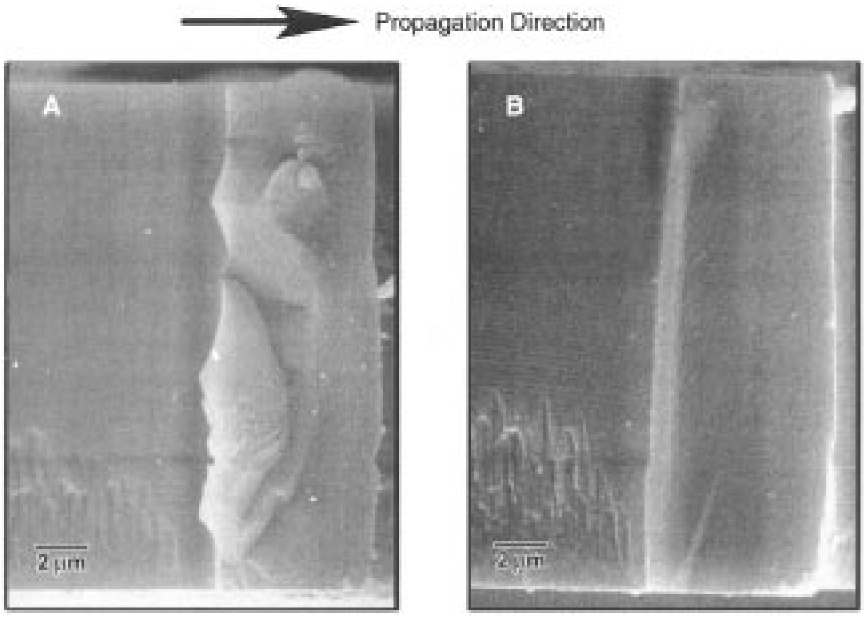

Fig. 6. Scanning electron micrographs of thin-film silicon specimen fracture surfaces with (a) short (20 s) and (b) long (48 days) lives. Propagation direction is from left to right as indicated by the arrow.

surface energy planes tend to propagate by cracking along multiple {111} cleavage planes [19]. In some cases, the initiation region and even the critical flaw can be identified.

The "long life" fracture surfaces of the thin-film silicon were significantly different from the short-life surfaces. The apparent initiation region of the longest life sample can be seen near the surface of the notch in Fig. 6(b) and is shown in detail in Fig. 7. The flaw, if present on the fracture surface, was too small to be imaged with the SEM. Compared to the large number of steps and ridges observed on the short life samples, the long life fracture surfaces are remarkably smooth. The failure proceeded on a (110) plane with the final failure's occurring on the (111) plane near the back edge of the sample. Small steps due to multiple {111} cracking are, however, seen at some points on the surface. The predominantly {110} crack path is unusual to observe in silicon and suggests that the active cracking mechanism under cyclic fatigue loading is different from that seen during quasi-static overload failure.

Fig. 7. Scanning electron micrograph of the initiation region of the "long life" sample shown in Fig. 6(b). The arrow indicates the direction of crack propagation.

C. Mechanisms

The results of this study strongly suggest that a distinct mechanism of failure is acting in single crystal silicon in room air under high-cycle fatigue loading conditions. The continuous compliance change, life-compliance correlation, and unique fracture surface morphology support this assertion. A variety of mechanisms, either individually or synergistically, may be responsible for the delayed failure of silicon. Although the evidence to date is insufficient to prove that a particular mechanism is operating, there are a few possibilities that are currently under investigation. Specifically, mechanisms associated with the loading conditions, the microstructure, and the environment are three possibilities that may account for the delayed failure of silicon observed in this study. These classes of mechanisms may be considered as those that would enable metal-like fatigue behavior and those that would be more consistent with fatigue mechanisms generally attributed to brittle materials.

The generation and motion of dislocations and the accumulation of plastic strain generally associated with fatigue in metals are not possible in silicon at low homologous temperatures. Proof of this may be found in the clear absence of dislocation activity at crack tips in silicon loaded in tension [29]. However, there is increasing evidence that very large compressive stresses can lead to plasticity at room temperature in silicon [30]; for example, the large hydrostatic stresses may suppress failure and allow the critical resolved shear stress of the material to be exceeded. Other explanations have been based on phase transformations in the presence of large compressive stresses. It is thus conceivable that the large compressive stresses in the vicinity of the notch could lead to localized flow and subsequent crack advance due to the plastic deformation, i.e., to a metal fatigue-type mechanism. If this is the case, then the magnitude of the load ratio, specifically, whether the loading is fully reversed or tension–tension, will have a significant influence on whether fatigue effects would occur in silicon films. Although the *S/N* behavior of the single crystal silicon films in this study may be "metal-like" in character, the authors believe that mechanisms associated with brittle material fatigue are more likely than room-temperature plasticity in silicon.

Brittle material fatigue is generally associated with the degradation of extrinsic toughening mechanisms, as discussed in the introduction. However, crack-tip shielding mechanisms associated with brittle material fatigue would not be expected to operate in single crystal silicon due to the absence of grain boundaries and the insignificant concentrations in secondary phases that would enable transformation toughening. We believe that it is more likely that the susceptibility of silicon-based oxides to stress corrosion cracking [31]–[33] is responsible for the fatigue behavior of single crystal silicon. In addition to the native oxide on the surface of silicon, oxides are also found in the bulk of the material [34]–[37]. These oxygen-rich phases may provide sites for crack initiation and preferential environmental attack. As noted in the Introduction, previous studies have shown that bulk silicon is insensitive to stress-corrosion cracking in moist air and water. However, under cyclic loading conditions, cracks in thin-film silicon grow faster in moist than in dry air [3], [4]. Whether this is a result of stress corrosion of the native oxide layer on the silicon, very slow stress-corrosion cracking of the silicon itself, or a corrosion fatigue mechanism is as yet unresolved. The feasibility of sustained-load tests that would clarify the existence of a stress-corrosion versus a true corrosion-fatigue effect is being explored. However, the progressive accumulation of damage in the sample and propagation of the crack along the (110) plane would be consistent with such environmental effects.

IV. Concluding Remarks

Silicon has been, and will continue to be, the dominant structural material for micromechanical components for years to come. Both the material and micromachining technology are readily available and comparatively inexpensive. For relatively low-stress applications such as pressure transducers and accelerometers, silicon is clearly a natural choice. Unfortunately, the convenience and success of silicon has bolstered the misconception that silicon is the ultimate in structural materials. The relatively high tensile strength and elastic modulus have eclipsed the fact that silicon is intrinsically brittle at room temperature. The materials science and mechanical engineering communities have come to terms with the limitations of bulk structural ceramics, including silicon. Unfortunately, the fact that critical structures must be designed for reliability, and not for strength, must be deemed as a critical concern for the MEMS community. Even more disturbing is the phenomenon described in these studies, namely, the prospect of time-delayed failure of silicon under cyclic loading conditions at lithographically patterned notches, as such alternating stresses and notches will inevitably be present in actual MEMS components.

V. Conclusions

Based on an experimental study of the cyclic fatigue of thin ($\sim$ 20 μm) films of single crystal silicon at high frequencies (40–50 kHz) in relatively moist room air, the following conclusions can be made.

1) Single crystal silicon thin films can degrade and fail under cyclic loading conditions in ambient air that contains water vapor at cyclic stresses some 50% of the single-cycle fracture strength.
2) The {110} crack path observed in the long-life ($> 10^6$ cycles) fatigue of single crystal silicon samples is distinct from the {111} paths seen for single-cycle overload fracture. This suggests that a mechanism other than the normal {111} cleavage is active during high-cycle fatigue in silicon.
3) The delayed failure of thin silicon films at stresses much lower than their fracture strength may be a significant limitation to the long-term frequency stability, reliability, and life of single crystal silicon micromechanical devices. Not only may these devices fail prematurely due to fatigue, but also the loss of stiffness may lead to a large accumulated error that may detrimentally affect components such as oscillators or gyroscopes that rely on a constant stiffness for timing or inertial measurement.

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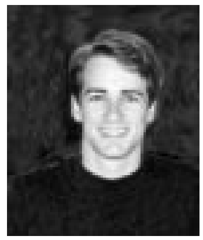

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